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4,5,6,7-Tetrachloro-2-(2-nitrophenyl)-isoindoline-1,3-dione

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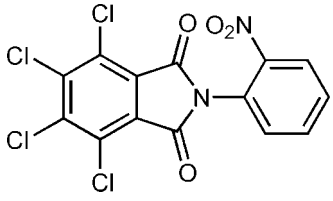
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 12.4.

In the title compound, $C_{14}H_4Cl_4N_2O_4$, the tetrachlorophthalimide unit and the nitrophenyl system are each essentially planar. The dihedral angle between the two planar systems is 74.6 (2)°.

Related literature

For related structures, see: Liang & Li (2006); Li *et al.* (2007). For background, see: Lima *et al.* (2002).



Experimental

Crystal data

$C_{14}H_4Cl_4N_2O_4$
M_r = 405.99
Triclinic, $P\overline{1}$
a = 7.4616 (13) Å
b = 8.8220 (16) Å
c = 13.137 (2) Å
α = 96.634 (2)°
β = 100.513 (2)°
γ = 113.368 (2)°
V = 763.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.80 mm^{-1}
T = 298 (2) K
0.47 × 0.39 × 0.23 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.706, T_{max} = 0.838
3929 measured reflections
2696 independent reflections
2449 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.04
2696 reflections
218 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2452).

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supplementary materials

Acta Cryst. (2007). E**63**, o3331 [**doi:10.1107/S1600536807030632**]

4,5,6,7-Tetrachloro-2-(2-nitrophenyl)isoindoline-1,3-dione

J. Li

Comment

Phthalimides and N-substituted phthalimides are an important class of compounds because of their interesting biological activities (Lima *et al.*, 2002). In this paper, the structure of the title compound, (I), is reported (Fig. 1). The geometry of molecule is close to the related compounds 2-(2-hydroxyphenyl)isoindoline-1,3-dione (Li *et al.*, 2007) and 2-(4-hydroxyphenyl)isoindoline-1,3-dione (Liang & Li, 2006). The tetrachlorophthalimide moiety system is essentially planar to within 0.019 (3) Å. The nitrophenyl system is planar to within 0.130 (2) Å. The dihedral angle between the nitrophenyl system and the tetrachlorophthalimide moiety is 74.6 (2) °. The crystal packing is shown in Fig. 2.

Experimental

A mixture of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (0.01 mol) and 2-nitrobenzenamine (0.01 mol) in acetic acid (10 ml) was refluxed for 1 h. After cooling, filtration and drying, the title compound was obtained: 10 mg was dissolved in 15 ml acetone, and the solution was kept at room temperature for 5 d. Natural evaporation gave colourless blocks of (I).

Refinement

The H atoms were initially located from difference maps, relocated in idealized locations (C—H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

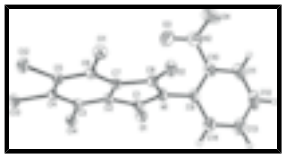

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).

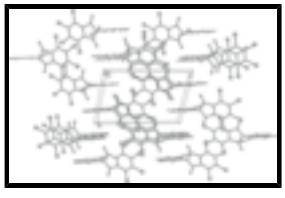

Fig. 2. The crystal packing of (I), viewed along the *a* axis.

4,5,6,7-Tetrachloro-2-(2-nitrophenyl)isoindoline-1,3-dione

Crystal data

$C_{14}H_4Cl_4N_2O_4$	$Z = 2$
$M_r = 405.99$	$F_{000} = 404$

Triclinic, $P\bar{1}$	D_x = 1.766 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.4616 (13) Å	Cell parameters from 3012 reflections
b = 8.8220 (16) Å	θ = 2.6–28.1º
c = 13.137 (2) Å	μ = 0.80 mm^{-1}
α = 96.634 (2)º	T = 298 (2) K
β = 100.513 (2)º	Block, colourless
γ = 113.368 (2)º	0.47 × 0.39 × 0.23 mm
V = 763.4 (2) Å^3	

Data collection

Bruker SMART CCD diffractometer	2696 independent reflections
Radiation source: fine-focus sealed tube	2449 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.013
T = 298(2) K	θ_{max} = 25.2º
ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −8→8
T_{min} = 0.706, T_{max} = 0.838	k = −4→10
3929 measured reflections	l = −15→14

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.2698P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.081	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.04	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2696 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
218 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.040 (3)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9838 (3)	0.6036 (2)	0.22533 (15)	0.0360 (4)
C2	1.0988 (3)	0.6611 (2)	0.33922 (14)	0.0316 (4)
C3	1.1670 (3)	0.5754 (2)	0.40646 (14)	0.0316 (4)
C4	1.2641 (3)	0.6587 (2)	0.51150 (14)	0.0339 (4)
C5	1.2918 (3)	0.8239 (2)	0.54681 (14)	0.0341 (4)
C6	1.2243 (3)	0.9100 (2)	0.47778 (14)	0.0327 (4)
C7	1.1267 (3)	0.8255 (2)	0.37398 (14)	0.0318 (4)
C8	1.0350 (3)	0.8813 (2)	0.28396 (15)	0.0364 (4)
C9	0.8483 (3)	0.7423 (3)	0.09699 (15)	0.0427 (5)
C10	0.6529 (3)	0.7315 (3)	0.07728 (16)	0.0478 (5)
C11	0.5585 (4)	0.7370 (3)	−0.02208 (19)	0.0660 (7)
H11	0.4290	0.7314	−0.0350	0.079*
C12	0.6573 (5)	0.7509 (4)	−0.1017 (2)	0.0785 (9)
H12	0.5939	0.7545	−0.1687	0.094*
C13	0.8482 (5)	0.7596 (4)	−0.0834 (2)	0.0763 (8)
H13	0.9130	0.7677	−0.1380	0.092*
C14	0.9444 (4)	0.7563 (4)	0.01582 (18)	0.0631 (7)
H14	1.0746	0.7636	0.0282	0.076*
N1	0.9562 (3)	0.7438 (2)	0.19898 (12)	0.0402 (4)
N2	0.5378 (3)	0.7075 (3)	0.15888 (16)	0.0602 (5)
Cl4	1.12983 (7)	0.37220 (6)	0.36281 (4)	0.04150 (15)
Cl1	1.26252 (7)	1.11402 (6)	0.52014 (4)	0.04185 (15)
Cl3	1.35075 (7)	0.55691 (7)	0.59779 (4)	0.04516 (16)
Cl2	1.41129 (8)	0.92336 (7)	0.67612 (4)	0.04823 (16)
O1	0.9219 (2)	0.47001 (18)	0.16717 (11)	0.0514 (4)
O2	1.0246 (2)	1.01248 (18)	0.28003 (11)	0.0512 (4)
O3	0.5849 (3)	0.6509 (3)	0.23320 (15)	0.0761 (5)
O4	0.3974 (3)	0.7486 (3)	0.14666 (18)	0.0993 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0380 (10)	0.0380 (10)	0.0333 (9)	0.0207 (8)	0.0035 (8)	0.0049 (8)
C2	0.0306 (9)	0.0327 (9)	0.0318 (9)	0.0147 (7)	0.0059 (7)	0.0069 (7)
C3	0.0275 (8)	0.0313 (9)	0.0376 (9)	0.0138 (7)	0.0076 (7)	0.0094 (7)
C4	0.0291 (9)	0.0408 (10)	0.0350 (9)	0.0164 (8)	0.0079 (7)	0.0151 (8)
C5	0.0286 (9)	0.0405 (10)	0.0296 (9)	0.0126 (8)	0.0046 (7)	0.0061 (8)
C6	0.0305 (9)	0.0317 (9)	0.0349 (9)	0.0123 (7)	0.0084 (7)	0.0061 (7)
C7	0.0315 (9)	0.0312 (9)	0.0340 (9)	0.0144 (7)	0.0082 (7)	0.0081 (7)

C8	0.0394 (10)	0.0378 (10)	0.0344 (9)	0.0195 (8)	0.0077 (8)	0.0079 (8)
C9	0.0595 (12)	0.0428 (11)	0.0299 (9)	0.0313 (10)	0.0005 (8)	0.0052 (8)
C10	0.0558 (13)	0.0462 (12)	0.0381 (11)	0.0281 (10)	−0.0037 (9)	−0.0026 (9)
C11	0.0772 (17)	0.0692 (16)	0.0478 (14)	0.0455 (14)	−0.0167 (12)	−0.0032 (12)
C12	0.127 (3)	0.0765 (18)	0.0360 (13)	0.0619 (18)	−0.0110 (14)	0.0062 (12)
C13	0.121 (3)	0.093 (2)	0.0369 (12)	0.0662 (19)	0.0196 (14)	0.0191 (13)
C14	0.0818 (17)	0.0836 (18)	0.0425 (12)	0.0530 (15)	0.0159 (12)	0.0165 (12)
N1	0.0508 (10)	0.0407 (9)	0.0307 (8)	0.0266 (8)	−0.0006 (7)	0.0046 (7)
N2	0.0490 (11)	0.0656 (13)	0.0533 (12)	0.0240 (10)	−0.0020 (9)	−0.0097 (10)
Cl4	0.0489 (3)	0.0346 (3)	0.0455 (3)	0.0235 (2)	0.0085 (2)	0.0093 (2)
Cl1	0.0447 (3)	0.0329 (3)	0.0438 (3)	0.0157 (2)	0.0078 (2)	0.0011 (2)
Cl3	0.0470 (3)	0.0535 (3)	0.0412 (3)	0.0275 (2)	0.0053 (2)	0.0195 (2)
Cl2	0.0503 (3)	0.0544 (3)	0.0314 (3)	0.0204 (2)	−0.0014 (2)	0.0024 (2)
O1	0.0643 (9)	0.0439 (8)	0.0411 (8)	0.0296 (7)	−0.0053 (7)	−0.0036 (7)
O2	0.0761 (10)	0.0418 (8)	0.0413 (8)	0.0347 (8)	0.0055 (7)	0.0099 (6)
O3	0.0786 (13)	0.0913 (14)	0.0582 (11)	0.0339 (11)	0.0187 (9)	0.0210 (10)
O4	0.0677 (13)	0.147 (2)	0.0871 (15)	0.0661 (15)	0.0022 (11)	−0.0070 (15)

Geometric parameters (Å, °)

C1—O1	1.191 (2)	C8—N1	1.395 (2)
C1—N1	1.402 (2)	C9—C14	1.380 (3)
C1—C2	1.498 (2)	C9—C10	1.396 (3)
C2—C3	1.381 (2)	C9—N1	1.427 (2)
C2—C7	1.388 (3)	C10—C11	1.382 (3)
C3—C4	1.395 (3)	C10—N2	1.475 (3)
C3—Cl4	1.7115 (18)	C11—C12	1.376 (4)
C4—C5	1.397 (3)	C11—H11	0.9300
C4—Cl3	1.7136 (18)	C12—C13	1.370 (4)
C5—C6	1.396 (3)	C12—H12	0.9300
C5—Cl2	1.7124 (18)	C13—C14	1.378 (3)
C6—C7	1.385 (3)	C13—H13	0.9300
C6—Cl1	1.7130 (19)	C14—H14	0.9300
C7—C8	1.489 (3)	N2—O3	1.205 (3)
C8—O2	1.196 (2)	N2—O4	1.228 (3)
?···?	?		
O1—C1—N1	125.26 (17)	C14—C9—C10	119.2 (2)
O1—C1—C2	130.10 (17)	C14—C9—N1	118.1 (2)
N1—C1—C2	104.62 (15)	C10—C9—N1	122.63 (19)
C3—C2—C7	121.50 (17)	C11—C10—C9	120.2 (2)
C3—C2—C1	130.08 (16)	C11—C10—N2	117.8 (2)
C7—C2—C1	108.39 (15)	C9—C10—N2	121.96 (18)
C2—C3—C4	117.91 (16)	C12—C11—C10	119.6 (3)
C2—C3—Cl4	121.00 (14)	C12—C11—H11	120.2
C4—C3—Cl4	121.08 (14)	C10—C11—H11	120.2
C3—C4—C5	120.73 (16)	C13—C12—C11	120.6 (2)
C3—C4—Cl3	119.49 (14)	C13—C12—H12	119.7
C5—C4—Cl3	119.78 (14)	C11—C12—H12	119.7
C6—C5—C4	120.85 (16)	C12—C13—C14	120.2 (3)

C6—C5—Cl2	119.28 (15)	C12—C13—H13	119.9
C4—C5—Cl2	119.88 (14)	C14—C13—H13	119.9
C7—C6—C5	117.85 (17)	C13—C14—C9	120.3 (3)
C7—C6—Cl1	121.17 (14)	C13—C14—H14	119.9
C5—C6—Cl1	120.98 (14)	C9—C14—H14	119.9
C6—C7—C2	121.16 (17)	C8—N1—C1	113.24 (15)
C6—C7—C8	130.23 (17)	C8—N1—C9	123.44 (16)
C2—C7—C8	108.60 (16)	C1—N1—C9	123.24 (16)
O2—C8—N1	124.59 (18)	O3—N2—O4	123.5 (3)
O2—C8—C7	130.28 (18)	O3—N2—C10	119.7 (2)
N1—C8—C7	105.12 (15)	O4—N2—C10	116.8 (2)
O1—C1—C2—C3	1.4 (4)	C6—C7—C8—N1	−179.26 (19)
N1—C1—C2—C3	179.82 (18)	C2—C7—C8—N1	0.0 (2)
O1—C1—C2—C7	−176.7 (2)	C14—C9—C10—C11	−0.8 (3)
N1—C1—C2—C7	1.8 (2)	N1—C9—C10—C11	177.6 (2)
C7—C2—C3—C4	0.5 (3)	C14—C9—C10—N2	176.8 (2)
C1—C2—C3—C4	−177.26 (18)	N1—C9—C10—N2	−4.8 (3)
C7—C2—C3—Cl4	179.48 (14)	C9—C10—C11—C12	0.9 (4)
C1—C2—C3—Cl4	1.7 (3)	N2—C10—C11—C12	−176.8 (2)
C2—C3—C4—C5	−0.3 (3)	C10—C11—C12—C13	−0.1 (4)
Cl4—C3—C4—C5	−179.26 (14)	C11—C12—C13—C14	−0.7 (4)
C2—C3—C4—Cl3	179.87 (13)	C12—C13—C14—C9	0.8 (4)
Cl4—C3—C4—Cl3	0.9 (2)	C10—C9—C14—C13	0.0 (4)
C3—C4—C5—C6	−0.4 (3)	N1—C9—C14—C13	−178.4 (2)
Cl3—C4—C5—C6	179.38 (14)	O2—C8—N1—C1	−178.3 (2)
C3—C4—C5—Cl2	179.95 (13)	C7—C8—N1—C1	1.3 (2)
Cl3—C4—C5—Cl2	−0.2 (2)	O2—C8—N1—C9	−1.6 (3)
C4—C5—C6—C7	0.9 (3)	C7—C8—N1—C9	177.93 (18)
Cl2—C5—C6—C7	−179.43 (13)	O1—C1—N1—C8	176.7 (2)
C4—C5—C6—Cl1	−178.69 (14)	C2—C1—N1—C8	−1.9 (2)
Cl2—C5—C6—Cl1	0.9 (2)	O1—C1—N1—C9	0.0 (3)
C5—C6—C7—C2	−0.7 (3)	C2—C1—N1—C9	−178.58 (18)
Cl1—C6—C7—C2	178.90 (14)	C14—C9—N1—C8	106.0 (2)
C5—C6—C7—C8	178.42 (18)	C10—C9—N1—C8	−72.4 (3)
Cl1—C6—C7—C8	−1.9 (3)	C14—C9—N1—C1	−77.6 (3)
C3—C2—C7—C6	0.0 (3)	C10—C9—N1—C1	103.9 (2)
C1—C2—C7—C6	178.22 (16)	C11—C10—N2—O3	157.2 (2)
C3—C2—C7—C8	−179.33 (16)	C9—C10—N2—O3	−20.5 (3)
C1—C2—C7—C8	−1.1 (2)	C11—C10—N2—O4	−23.3 (3)
C6—C7—C8—O2	0.3 (4)	C9—C10—N2—O4	159.0 (2)
C2—C7—C8—O2	179.5 (2)		

Fig. 1

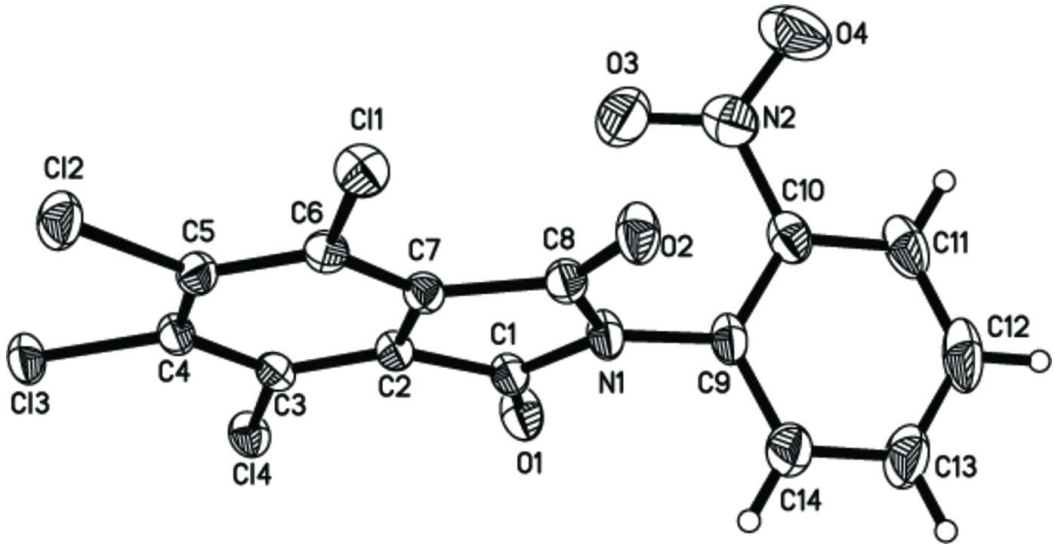
O4
O3
N2
Cl1
Cl2
C10
C6
C5
C7
C8
O2
C11
C12
C1
C4
N1
C9
Cl3
C2
C3
O1
Cl4
C13
C14

Fig. 2